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UNION CARBIDE

SECOND QUARTERLY REPORT

SECONDARY ZINC-OXYGEN CELL FOR SPACECRAFT APPLICATIONS

(23 SEPT., 1966 - 23 DEC., 1966)

CONTRACT NO. NAS-5-10247
for
GODDARD SPACE FLIGHT CENTER
GREENBELT, MARYLAND

Prepared by

UNION CARBIDE CORPORATION
CONSUMER PRODUCTS DIVISION
RESEARCH LABORATORY-PARMA, OHIO

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APPROVED BY:

R. A. Powers, Director

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ABSTRACT

The test program to date has shown that the zinc-oxygen system is rechargeable with good voltage regulation over a range of cycle regimes including two-hour discharge/two-hour charge, two-hour discharge/six hour charge, and twenty-four hour discharge/twenty-four hour charge. In the two-hour discharge/two hour charge cycle, over 50 cycles have been obtained above 1.0 volt terminal discharge voltage at a zinc depth of 14 percent with 10 ampere-hour cells. Deeper zinc discharge depth to 28 percent of theoretical has been demonstrated with the twenty-four hour discharge/twenty-four hour charge cycle.

Use of a membrane type C-3 separator produced by Borden Chemical Company under contract for NASA was a factor in achieving the aforementioned cycle life on the two-hour discharge/two-hour charge regime, even though the cell was cycled with abusive overcharge and only a single layer of C-3 separator was used. A re-design of the unit test cell so as to seal the zinc electrode behind the C-3 film separator, in a chamber inaccessible to oxygen bubbles, effectively prevents oxygen from "chemically shorting" the cell. The cells are operated in a "flooded condition" with a nickel charging electrode being used to prevent oxidation of carbon catalyst in the "fixed-zone" oxygen electrode. The use of more than one layer of C-3 separator in conjunction with the mechanical cushion non-woven separator on either side of the C-3 is expected to lead to cell cycle life several times beyond that obtained to date.

While the major effort directed toward system optimization is being conducted at room temperature (25°C) cycling performance evaluations at both 0°C and 40°C have been initiated and will be continued. As of this time the cells have undergone 4 two-hour discharge/two-hour charge cycles at 0°C, and 9 cycles at 40°C at a discharge depth of 14 percent.

* * * *

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INTRODUCTION

Work conducted to date has demonstrated the feasibility of recharging the zinc-oxygen battery system. The technical approach used was based upon Union Carbide's background experience in fuel cells, Air Cells, and rechargeable battery system technology.

The testing of unit cells has proved that cycle regimes of 2 hours discharge/ 2 hours charge, 2 hours discharge/6 hours charge, 24 hours discharge/24 hours charge are practical with good discharge voltage regulation. In the case of the 2 hour discharge/2 hour charge, over 50 cycles have been obtained above the 1.0V terminal discharge voltage at a zinc depth of 14 percent of the theoretical 10 ampere hours (12.1 grams of zinc). Deeper discharge depths to 28 percent of the theoretical zinc capacity have been attained on 24 hour discharge/24 hour charge test regime.

A major contributor toward achieving the aforementioned cycle life was the use of a membrane type C-3 separator produced by Borden Chemical Company under contract for NASA. This separator effectively isolates soluble zincate in the neighborhood of the zinc electrode so that zinc treeing is minimized. Even greater improvements in cycle life are expected through the use of multiple layers of this separator.

DISCUSSION

A. Experimental Unit Cell Construction

The construction of the experimental unit cell used to investigate the zinc oxygen system is that shown in Figure 1. A detailed description of cell components is given in Table I. The cell is built by enclosing the oxygen cathode within a cast "Lucite" frame (L) patterned after the cell assembly techniques that have been developed over a period of years in the company's fuel cell work. An oxygen gas inlet and outlet are provided shown as (J) and (K). These ports communicate to the region of the porous nickel back side of the oxygen electrode interface between (H) and (G). A polypropylene screen separator (F) is used to separate the catalyzed surface of the oxygen cathode from the nickel screen charging electrode (E). A similar polypropylene screen (D) separates the other side of the charging electrode from the separator (C) of the zinc electrode. This separator is a membrane film (C-3) produced by the Borden Chemical Company under contract fot NASA (1, 2). The zinc electrode (B) which completes the cell is enclosed in a basket of expanded silver metal that is spot welded to a silver sheet collector (A). The C-3 film separator is sealed to the "Lucite" frame in such a manner that the chamber holding the zinc electrode is inaccessible to oxygen bubbles that are formed at the charging electrode during cell charge. In this manner "chemical shorting" is effectively prevented. The test cells are operated in flooded condition during both discharge and charge. Oxidation of carbon catalyzed layer on the surface of the oxygen cathode is prevented during charging through the use of the nickel screen charging electrode.

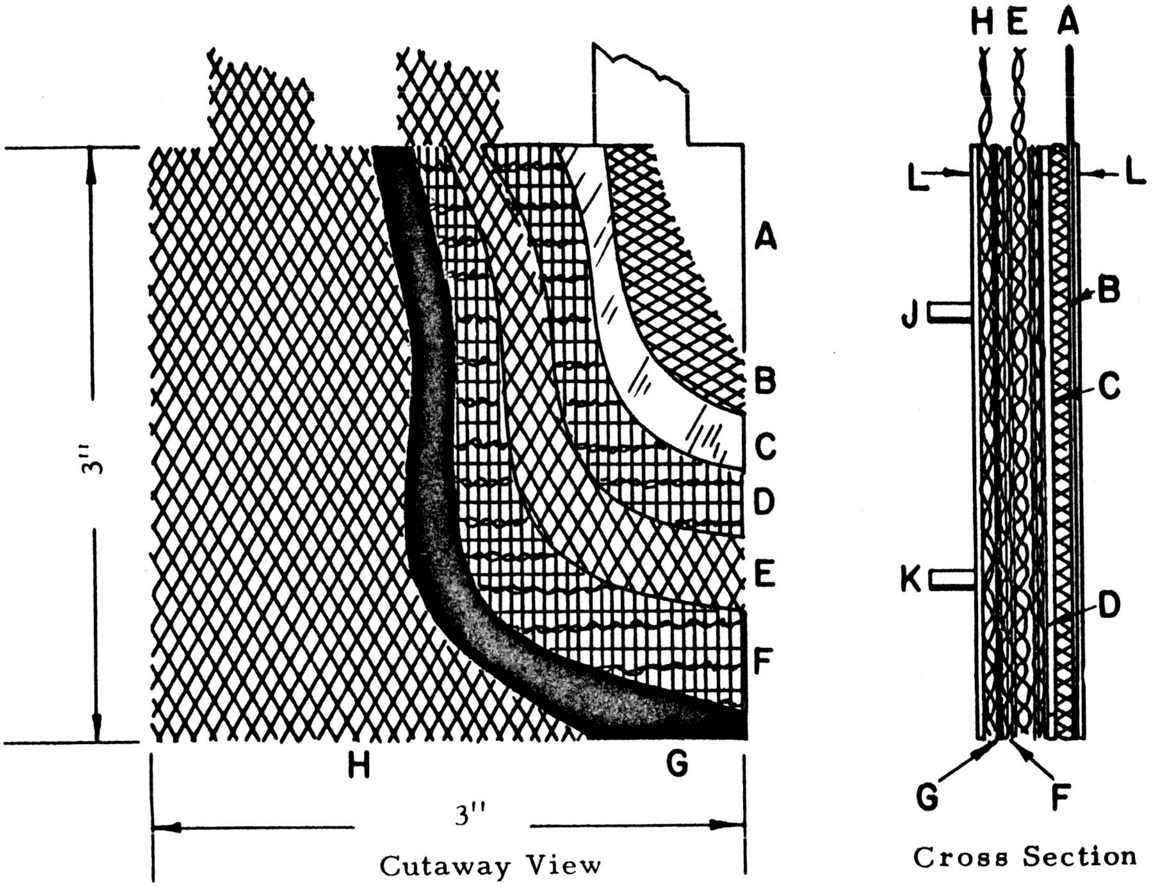
TABLE I
COMPONENTS OF EXPERIMENTAL UNIT CELL

	Dimensions	Weight (g)
"Fixed-Zone" Oxygen Electrode[a]	3" x 3" x 0.022"	10.0
Polypropylene Screen[b]	3" x 3" x 0.030"	1.5
Expanded Nickel Metal[c] Charging Electrode	3" x 3" x 0.020"	2.6
Polypropylene Screen[b]	3" x 3" x 0.030"	1.5
C-3 Membrane Separator (dry)[d]	3" x 3" x 0.0015"	0.3
Zinc Electrode[a]	3" x 3" x 0.100"	12.2
Electrolyte (44% KOH + 2% ZnO dissolved therein)		25.0
Oxygen Source[e]	oxygen cylinder	

[a] UCC Type 2 electrode
[b] Lamport
[c] Exmet
[d] Borden
[e] Linde

FIGURE 1

CUTAWAY AND CROSS SECTION VIEWS OF EXPERIMENTAL ZINC-OXYGEN RECHARGEABLE UNIT CELL



A	Silver sheet anode collector
B	Zinc anode restrained in a silver screen envelope
C	Separator
D	Polypropylene screen
E	Nickel Screen (Charging Electrode)
F	Polypropylene screen
G	"Fixed-Zone" oxygen electrode
H	Nickel screen cathode collector
J	Gas inlet
K	Gas Outlet
L	"Lucite" backing plates

B. Description of Electrodes

1. Oxygen Electrode

The oxygen electrode employed is the thin "fixed-zone" type that is based upon the use of an active carbon layer on one side of a thin but strong porous metal backing. This electrode was developed by Union Carbide over a period of time as an electrode combining high-rate capability with low cost (3-5). When oxygen is evolved electrochemically from a carbon-metal duplex electrode, the carbon is oxidized to form soluble quinone type compounds, it then loses its hydrophobic characteristics and the electrode becomes susceptible to flooding. A feature in the present cell is the use of a separate nickel charging electrode which is in the circuit during charging in the place of the oxygen electrode and upon which the oxygen is evolved. In this manner oxidation of the carbon catalyzed layer on the oxygen cathode is avoided during charging. Thus the reversible oxygen electrode is truly the combination of oxygen fuel cell electrode and a separate nickel screen charging electrode.

Work to date has shown that the use of a separate nickel screen charging electrode which is used exclusively for oxygen evolution in the charging mode prevents the degradation of the oxygen cathode and renders the rechargeable zinc-oxygen system feasible.

In practice the nickel screen is placed between the zinc anode and the oxygen cathode and is separated from each by a polypropylene separator. During charge, the oxygen cathode is removed from the circuit and oxygen is evolved only from the nickel screen charging electrode, while oxidized zinc is being reduced at the zinc electrode. During discharge the oxygen cathode is connected to the circuit with the zinc anode in normal fashion and the charging electrode is removed from the circuit.

2. Rechargeable Zinc Electrodes

The zinc electrode, as previously developed at Union Carbide, is made by folding an expanded silver sheet in envelope fashion so as to enclose the active zinc material. This unit is next pressed and spot-welded to a silver backing sheet. Finally it is sealed into the plastic cell frame in such a way that the active zinc is protected by a C-3 membrane separator from oxygen evolved at the charging electrode.

C. Description of Membrane Separator

Use of a membrane type (C-3) separator produced by Borden Chemical Company under contract for NASA has contributed substantially to improved cycle life. Even when only a single layer of C-3 separator* film was used, 50 cycles of 2 hour discharge/2 hour charge were obtained above a terminal discharge voltage of 1.0 volts. This performance was obtained although an abusive 50 percent overcharge was used in the cycling regime and cell operation was essentially unattended. The use of multiple layers of C-3 separators in conjunction with a protective non-woven cushion separator on either side of C-3 is expected to substantially extend cycle life beyond 50 cycles.

The C-3 Borden film is reported to be a composition of 30 percent poly(vinyl methyl ether/maleic anhydride) in a methyl cellulose base. Such a separator has a closely controlled pore size which helps prevent the growth of zinc dendrites through the separator. This separator is further stated to be particularly resistant to oxidation, an important advantage in a zinc-oxygen rechargeable cell. The C-3 membrane effectively separates the anolyte from the catholyte thereby improving the cycle life of the cell. Soluble zincate is thus kept in the anolyte in the vicinity of the zinc electrode, so that conditions for forming of zinc trees through the separator are minimized. This film type separator of controlled pore size further minimizes the possibility of oxygen bubbles formed during charging from reaching the zinc electrode and thereby "chemically shorting" the cell.

D. Rechargeable Unit Cell Performance

1. Performance at 25°C

Unit cell performance is being evaluated on both the 2 hour discharge/

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*electrolyte wet thickness = 0.0023"

2 hour charge and the 24 hour discharge/24 hour charge cycle modes. Fifty 2 hour discharge/2 hour charge cycles have been obtained above a terminal discharge voltage of 1.0 volt at 14 percent zinc depth based upon the theoretical ampere-hour equivalents of actual weight of zinc used. This performance was obtained under abusive conditions of 50 percent overcharge while the cell operated unattended with only a single layer of C-3 separator. Cycle life improvement to several hundred cycles is anticipated when multiple layers of C-3 separator are used in conjunction with a balanced charge-discharge cycle mode.

Figure 2 presents the discharge curves obtained over the first 50 abuse cycles during the operation of this cell. Table II summarizes cell performance for these 50 cycles.

Figure 3 is a graph of the cell voltage at the end of each 2 hour discharge over the same 50 cycle period.

Figures 4 through 9 present the 1st, 10th, 20th, 30th, 40th, and 50th two hour discharge on a large scale.

The unit cell operation at 25°C on the 24 hour discharge/24 hour charge cycle mode is presented in Figures 10 through 12, respectively, for the 1st, 8th, and 14th twenty-four hour discharge.

Figures 13 and 14 show the charge curves as well as the discharge curves for cells operating at 25°C at each of the 2 hour discharge/2 hour charge and 24 hour discharge/24 hour charge regimes. Both O_2 to Zn as well as Ni (charging electrode) to Zn potentials are plotted.

2. <u>Performance at 0°C</u>

Cell performance at 0°C on the 2 hour discharge/2 hour charge cycle has been initiated. Discharge curves for the 1st and 4th cycles are presented in Figures 15 and 16.

3. <u>Performance at 40°C</u>

Unit cell performance at 40°C has been conducted on the 2 hour discharge/2 hour charge cycle through 9 cycles thus far. The discharge curves obtained on the 1st, 5th and 9th cycles are presented in Figure 17 through 19.

FIGURE 2

DISCHARGE OF RECHARGEABLE ZINC-OXYGEN UNIT CELL FOR FIRST 50 CYCLES ON 2 HOUR DISCHARGE/2 HOUR CHARGE REGIME AT 25°C

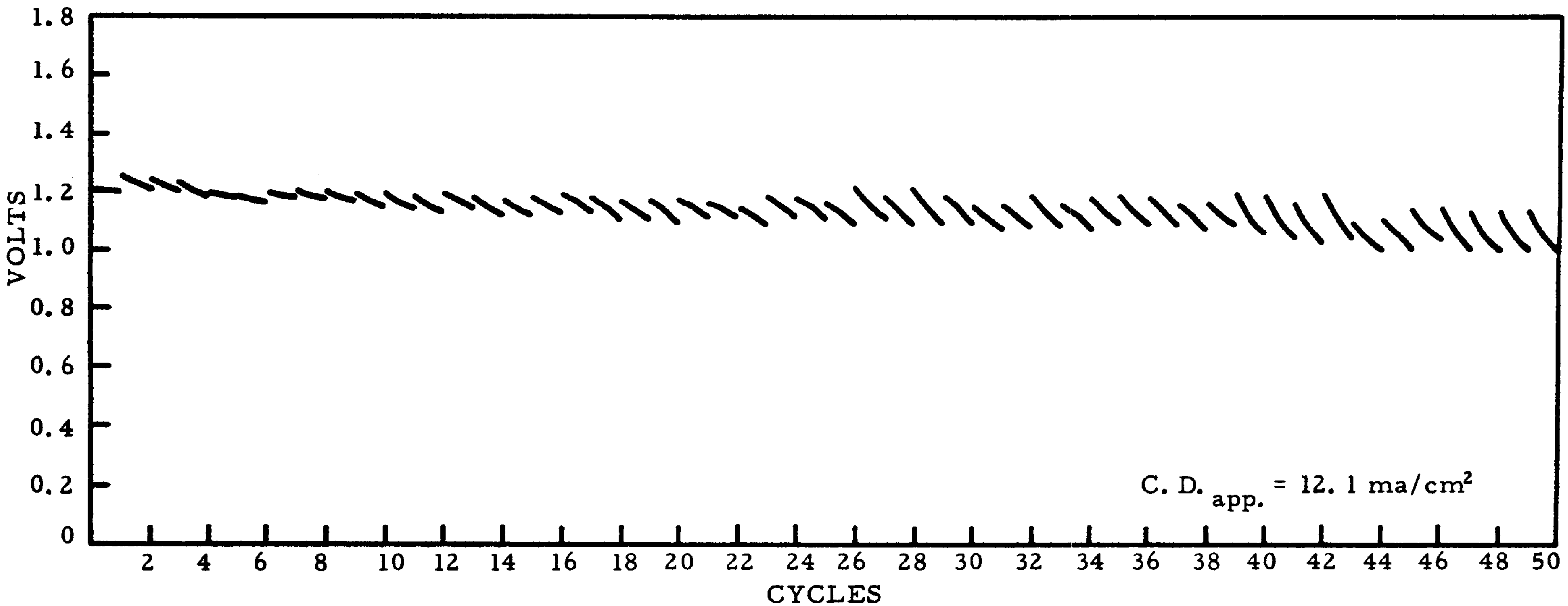

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TABLE II

PERFORMANCE ANALYSIS OF RECHARGEABLE ZINC-OXYGEN UNIT CELL

2 Hour Discharge - 2 Hour Charge (Plus 50% overcharge)

25°C

Discharge Number	Avg. Voltage (Volts)	Ave. Current (amperes)	Avg. Apparent Current Density ma/cm^2	Amp-Hrs Delivered
1	1.21	0.69	11.9	1.38
2	1.23	0.70	12.0	1.40
3	1.22	0.70	12.0	1.40
4	1.20	0.69	11.8	1.38
5	1.18	0.67	11.5	1.34
6	1.17	0.67	11.5	1.34
7	1.18	0.67	11.5	1.34
8	1.18	0.67	11.5	1.34
9	1.17	0.67	11.5	1.34
10	1.16	0.66	11.4	1.32
11	1.15	0.66	11.4	1.32
12	1.14	0.65	11.2	1.30
13	1.16	0.66	11.4	1.32
14	1.15	0.66	11.4	1.32
15	1.14	0.65	11.2	1.30
16	1.15	0.66	11.4	1.32
17	1.16	0.66	11.4	1.32
18	1.14	0.65	11.2	1.30
19	1.14	0.65	11.2	1.30
20	1.14	0.65	11.2	1.30
21	1.15	0.66	11.4	1.32
22	1.14	0.65	11.2	1.30
23	1.12	0.64	11.0	1.28
24	1.14	0.65	11.2	1.30
25	1.15	0.66	11.4	1.32

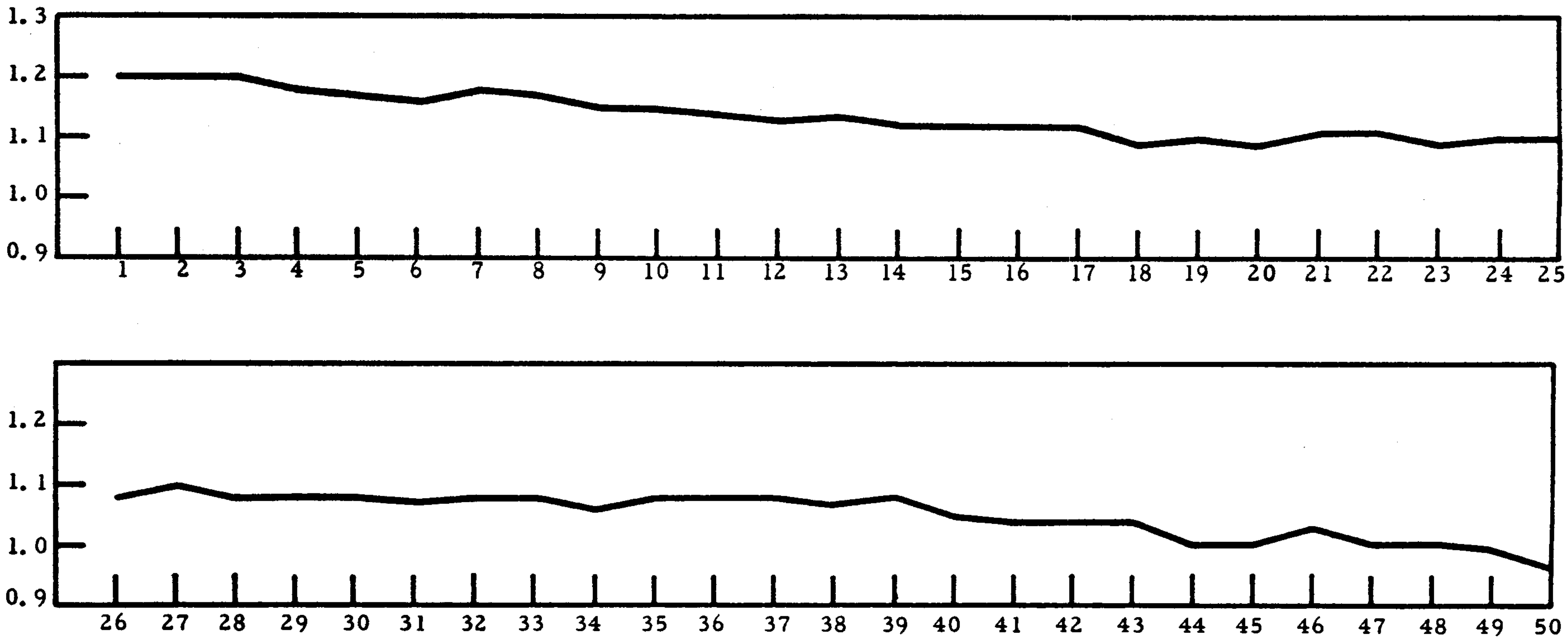
Continued

TABLE II-- Concluded

Discharge Number	Avg. Voltage (Volts)	Avg. Current (amperes)	Avg. Apparent Current Density ma/cm^2	Amp-Hrs Delivered
26	1.13	0.65	11.2	1.30
27	1.13	0.65	11.2	1.30
28	1.13	0.65	11.2	1.30
29	1.14	0.65	11.2	1.30
30	1.14	0.65	11.2	1.30
31	1.12	0.64	11.0	1.28
32	1.12	0.64	11.0	1.28
33	1.12	0.64	11.0	1.28
34	1.12	0.64	11.0	1.28
35	1.12	0.64	11.0	1.28
36	1.12	0.64	11.0	1.28
37	1.14	0.65	11.2	1.30
38	1.12	0.64	11.0	1.28
39	1.11	0.63	10.8	1.26
40	1.09	0.62	10.7	1.24
41	1.10	0.63	10.8	1.26
42	1.08	0.61	10.5	1.22
43	1.10	0.63	10.8	1.26
44	1.03	0.59	10.2	1.18
45	1.06	0.61	10.5	1.22
46	1.07	0.61	10.5	1.22
47	1.05	0.60	10.3	1.20
48	1.04	0.59	10.2	1.18
49	1.03	0.59	10.2	1.18
50	1.02	0.58	10.0	1.16

FIGURE 3

TERMINAL DISCHARGE VOLTAGE OF RECHARGEABLE ZINC-OXYGEN UNIT CELL
2 HOUR DISCHARGE/ 2 HOUR CHARGE (Plus 50% Overcharge)
25°C



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4. Comparison of 0°, 25°, and 40°C Performance

Comparison of unit cell performance on 2 hour discharge/2 hour charge cycle for the first four cycles at the three temperatures tested, 0°, 25°, and 40°C are shown in Figures 20 through 23. Examination of these figures reveals that discharge occurs at a higher voltage level at the higher temperature.

NEW TECHNOLOGY

There are no new technological advances falling within the scope of this contract to be reported at this time.

PROGRAM FOR NEXT REPORTING INTERVAL

Efforts will be concentrated on extending the cycle life of the present zinc-oxygen cell. Further modification of the unit test cell together with the use of multiple layers of Borden film type separator (C-3) will be explored. The use of a mechanical cushioning non-woven separator on either side of the C-3 films will be examined in an effort to protect the C-3 separator from mechanical puncture either from the zinc side or from the charging electrode side. Such improved cells are expected to provide even greater cycle life, substantially extending the present 50 cycles obtained on the 2 hour discharge/2 hour charge regime. Test cell evaluations of the improved cells constructed in accordance with the above will be evaluated over the 0° to 40°C temperature range of interest to NASA.

Cell tests will be conducted at 0°, 25°, and 40°C on both the 2 hour discharge/2 hour charge and 24 hour discharge/24 hour charge test regimes. The overall anticipated effort for the next reporting period will be concerned with further unit cell optimization. This will include emphasis upon (a) increasing cell cycle life, (b) optimizing cell design and minimizing required cell components from a weight and volume standpoint, and (c) increasing the depth of cell discharge.

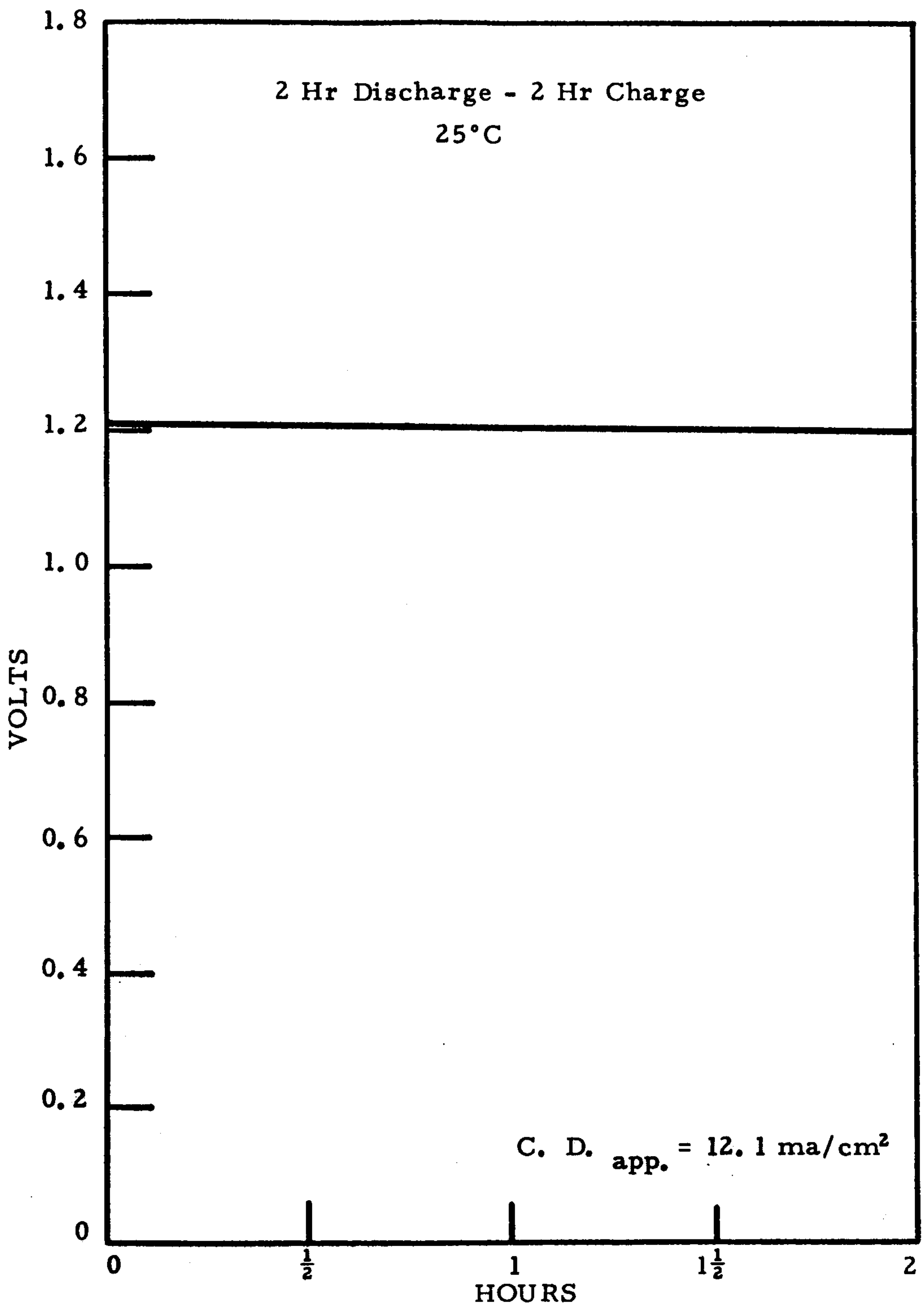
CONCLUSIONS AND RECOMMENDATIONS

Work conducted during the present quarter has shown that the development of a rechargeable zinc-oxygen system operating with good voltage regulation for at least 50 cycles is possible. The best results were obtained with a film type separator (C-3) made by the Borden Chemical Company under contract for NASA. This cycle life was obtained on a 2 hour discharge/2 hour charge regime at 25°C in a cell containing only a single layer of C-3 separator and which was operated at a 50 percent abusive overcharge. It is recommended that the use of multiple layers of C-3 separator in cells operating with a balanced charge-discharge test be explored. It is reasonable to expect further extensions of cycle life may result from such changes. It is recommended that such improved cells be cycled at the three temperatures of 0°, 25°, and 40°C on both the 2 hour discharge/2 hour charge and 24 hour discharge/24 hour charge test regimes in order to more completely bracket the temperature range and test regimes of interest to NASA.

Cell discharge conducted during the present quarter was taken to the 14 percent zinc depth based on the theoretical equivalent of the actual weight of zinc used in the case of the 2 hour discharge/2 hour charge regime, and to the 28 percent zinc depth in the case of the 24 hour discharge/24 hour charge regime. It is recommended deeper discharges up to the 50 percent depth be investigated during the next quarter.

FIGURE 4

1ST DISCHARGE OF RECHARGEABLE $Zn-O_2$ UNIT CELL



C-3310

FIGURE 5

10TH DISCHARGE OF RECHARGEABLE $Zn-O_2$ UNIT CELL

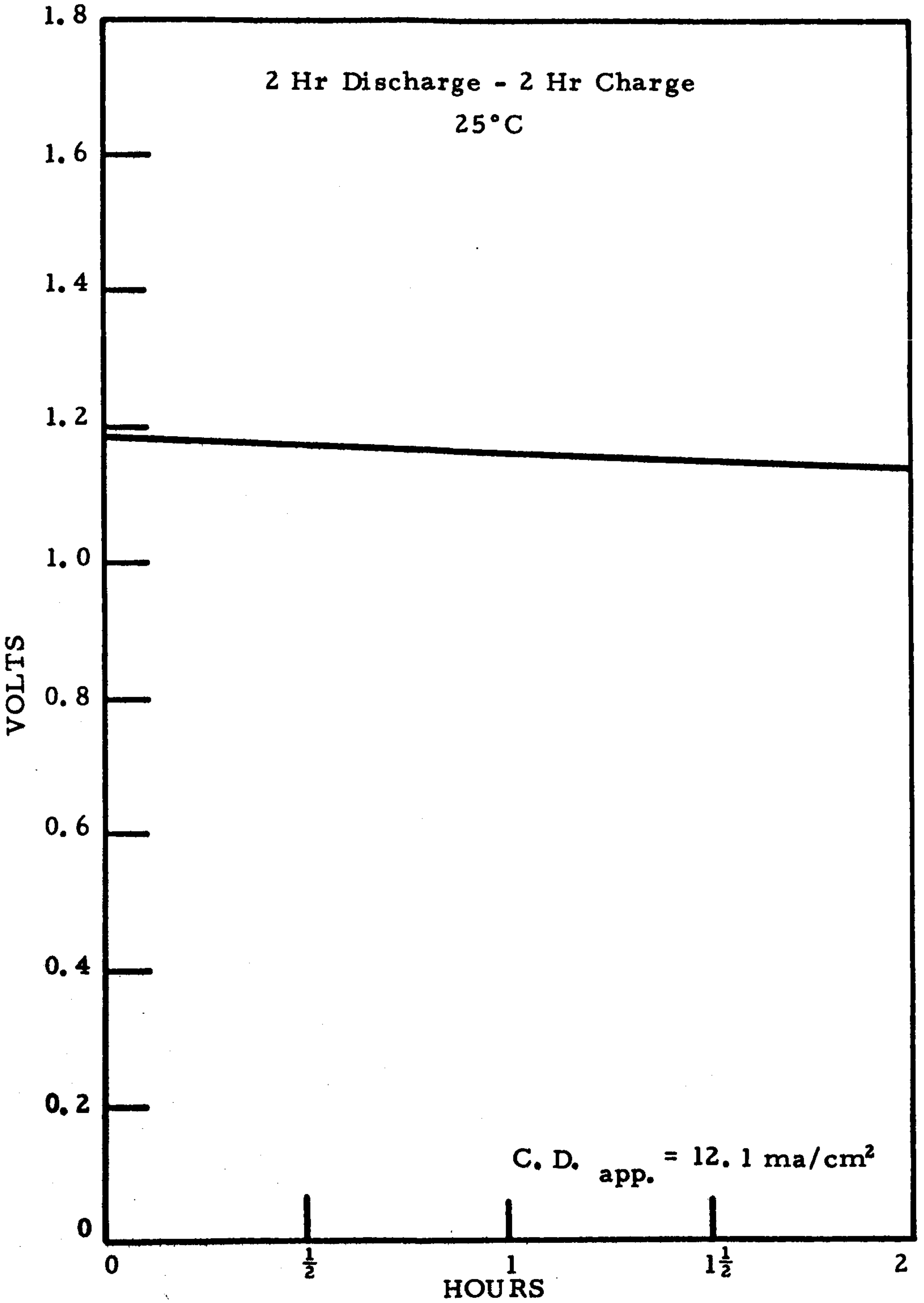
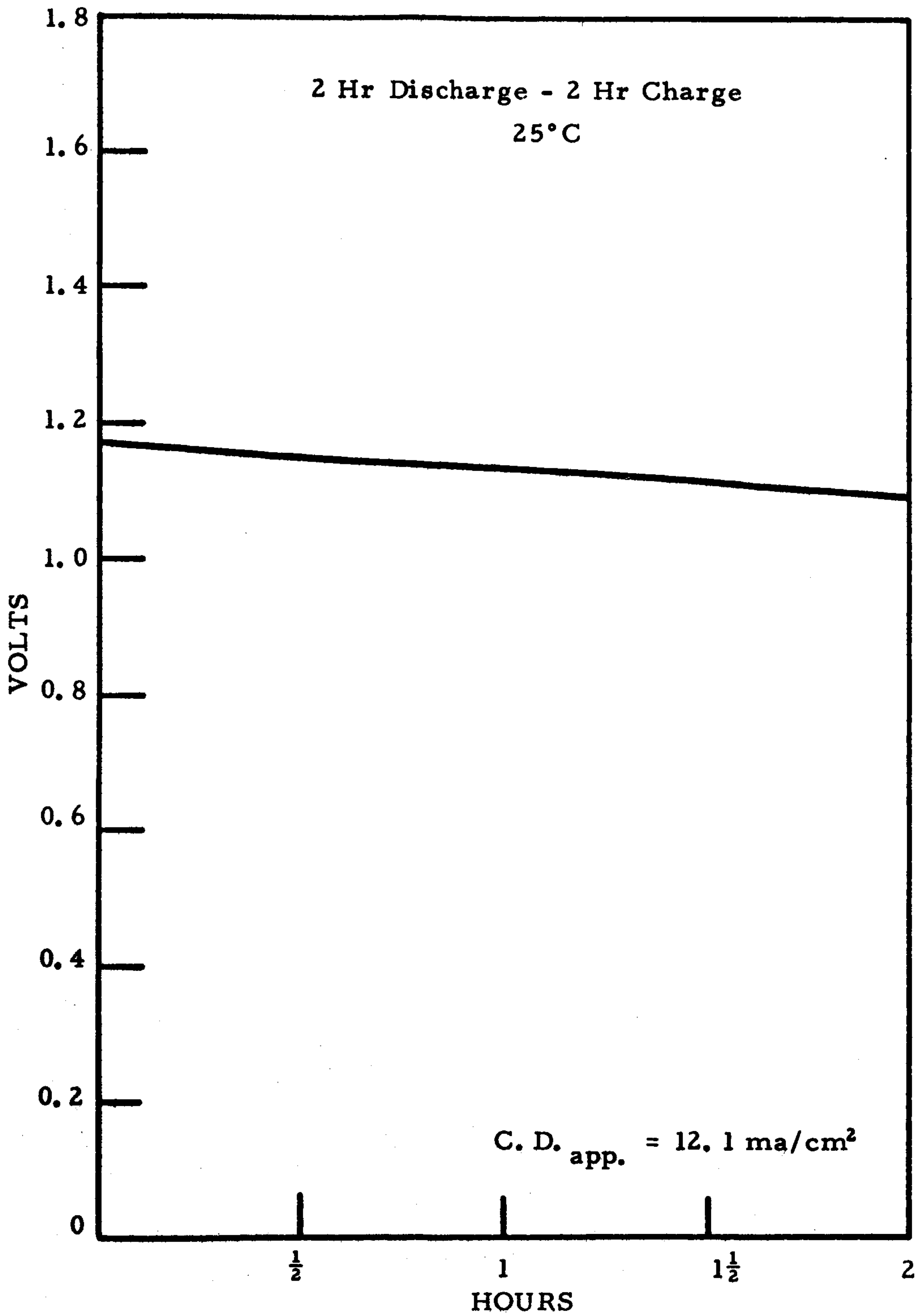


FIGURE 6

20TH DISCHARGE OF RECHARGEABLE $Zn-O_2$ UNIT CELL



C-3314

FIGURE 7

30TH DISCHARGE OF RECHARGEABLE $Zn-O_2$ UNIT CELL

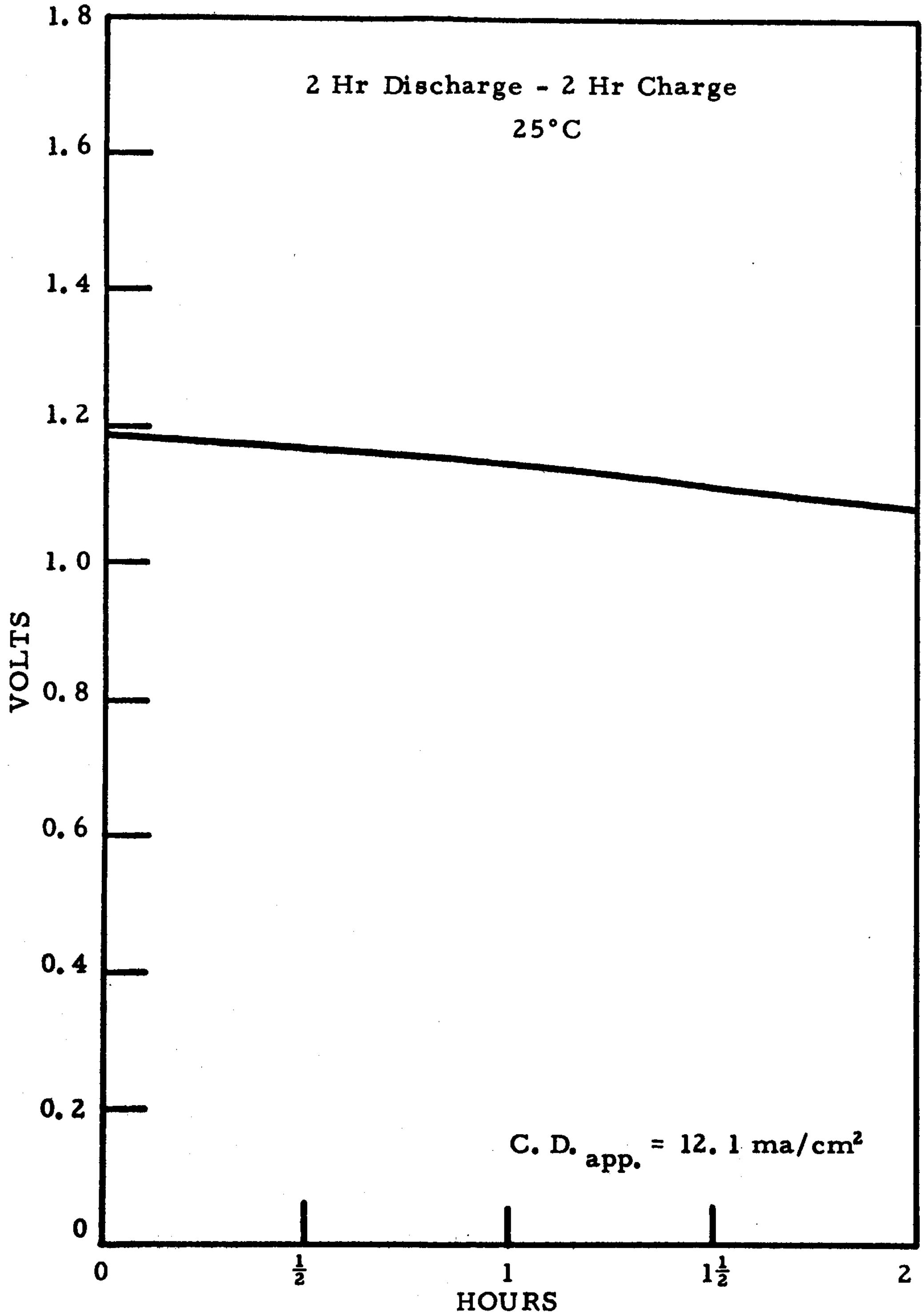


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FIGURE 8

40TH DISCHARGE OF RECHARGEABLE $Zn-O_2$ UNIT CELL

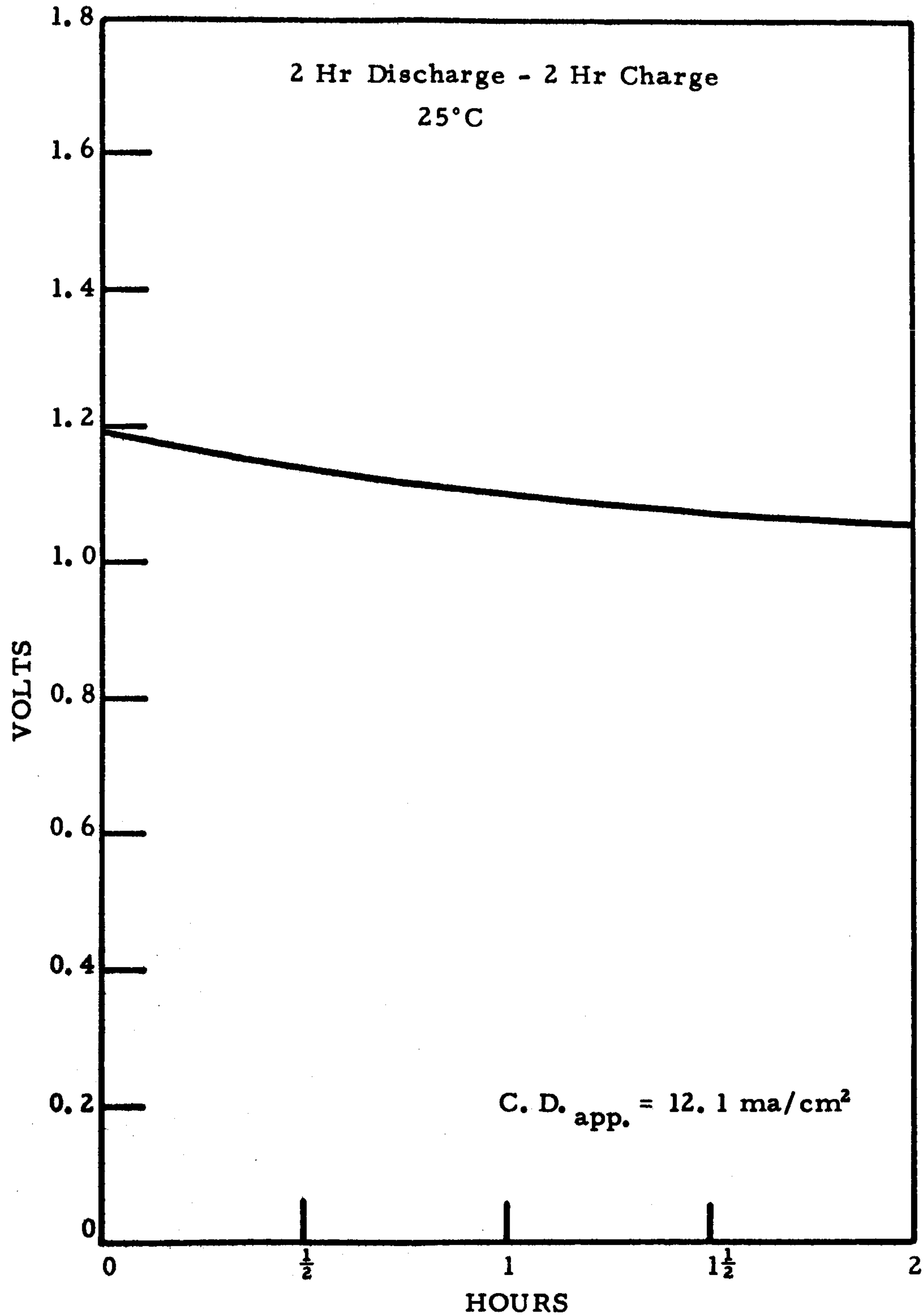

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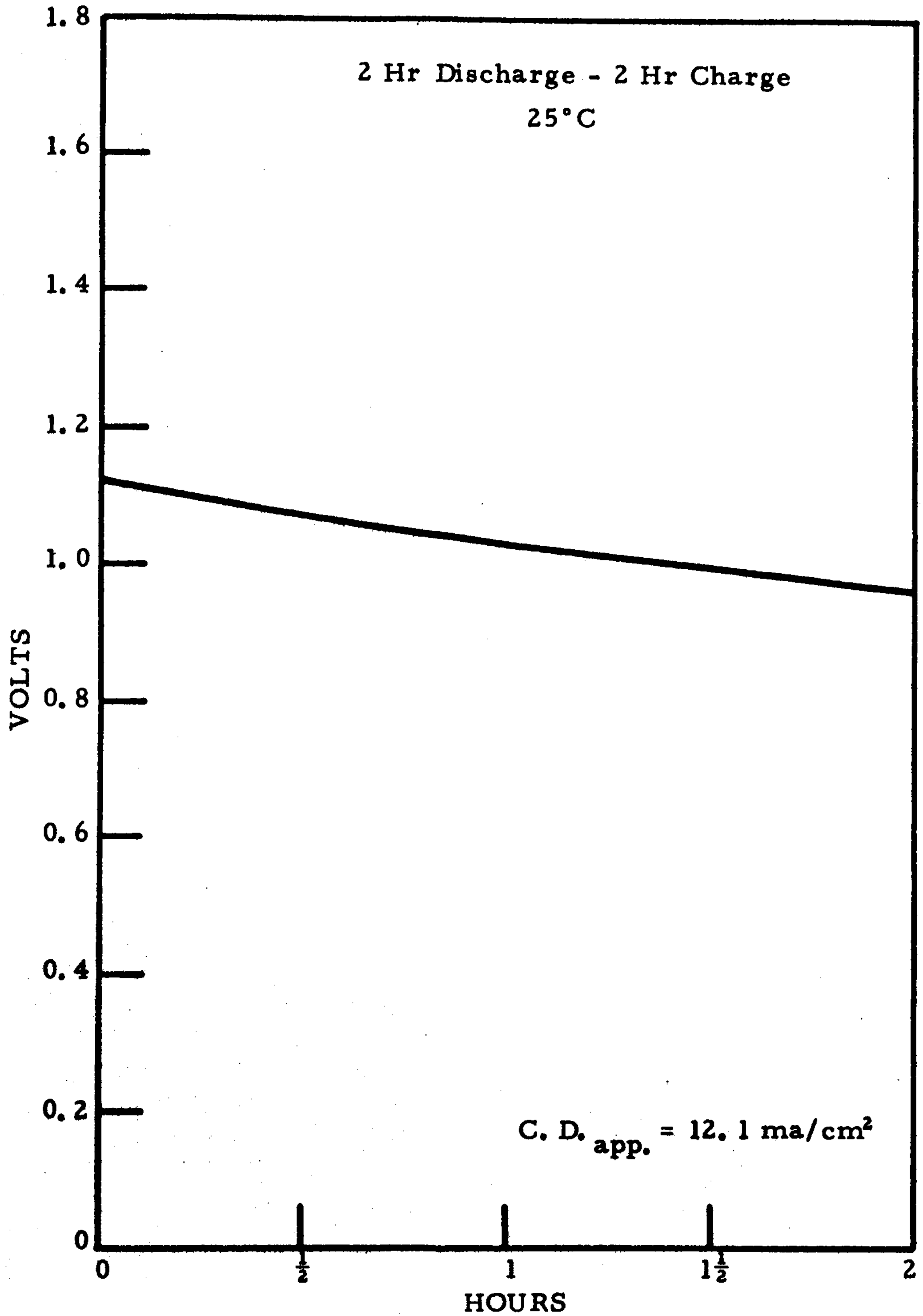
FIGURE 9

50TH DISCHARGE OF RECHARGEABLE $Zn-O_2$ UNIT CELL



C-3316

FIGURE 10

1ST DISCHARGE OF RECHARGEABLE $Zn-O_2$ UNIT CELL

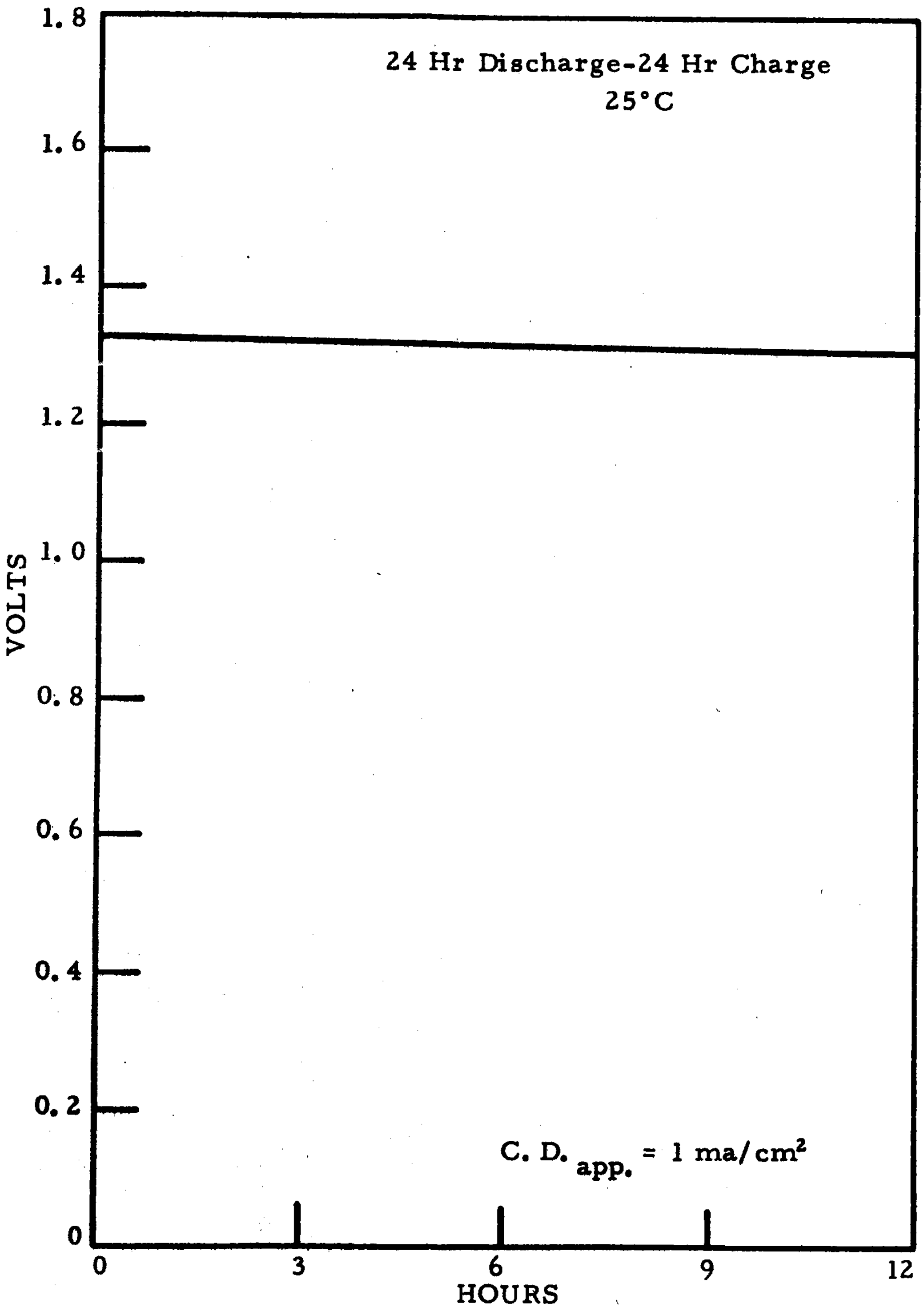


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FIGURE 11

8TH DISCHARGE OF RECHARGEABLE $Zn-O_2$ UNIT CELL

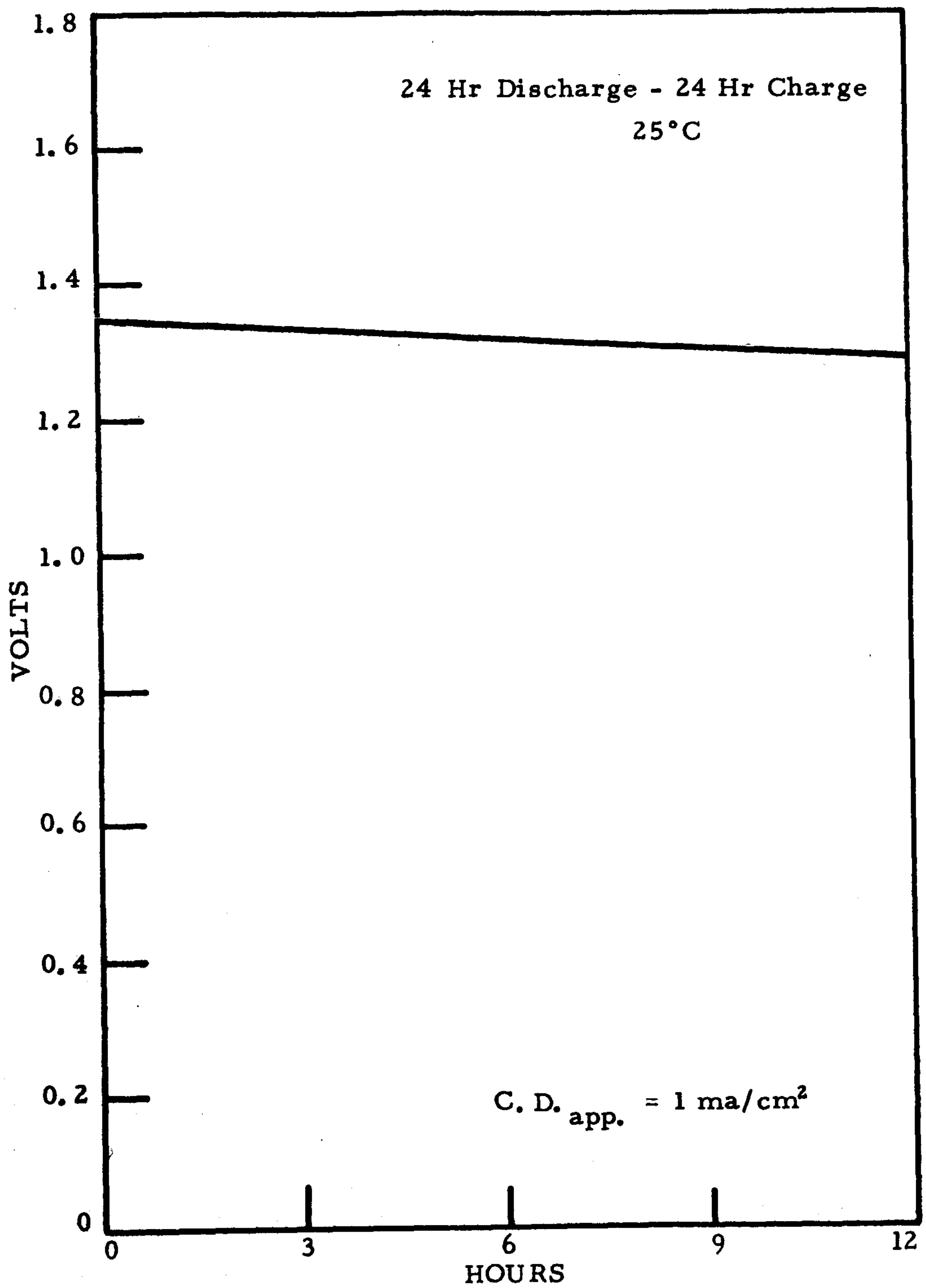

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FIGURE 12

14TH DISCHARGE OF RECHARGEABLE $Zn-O_2$ UNIT CELL

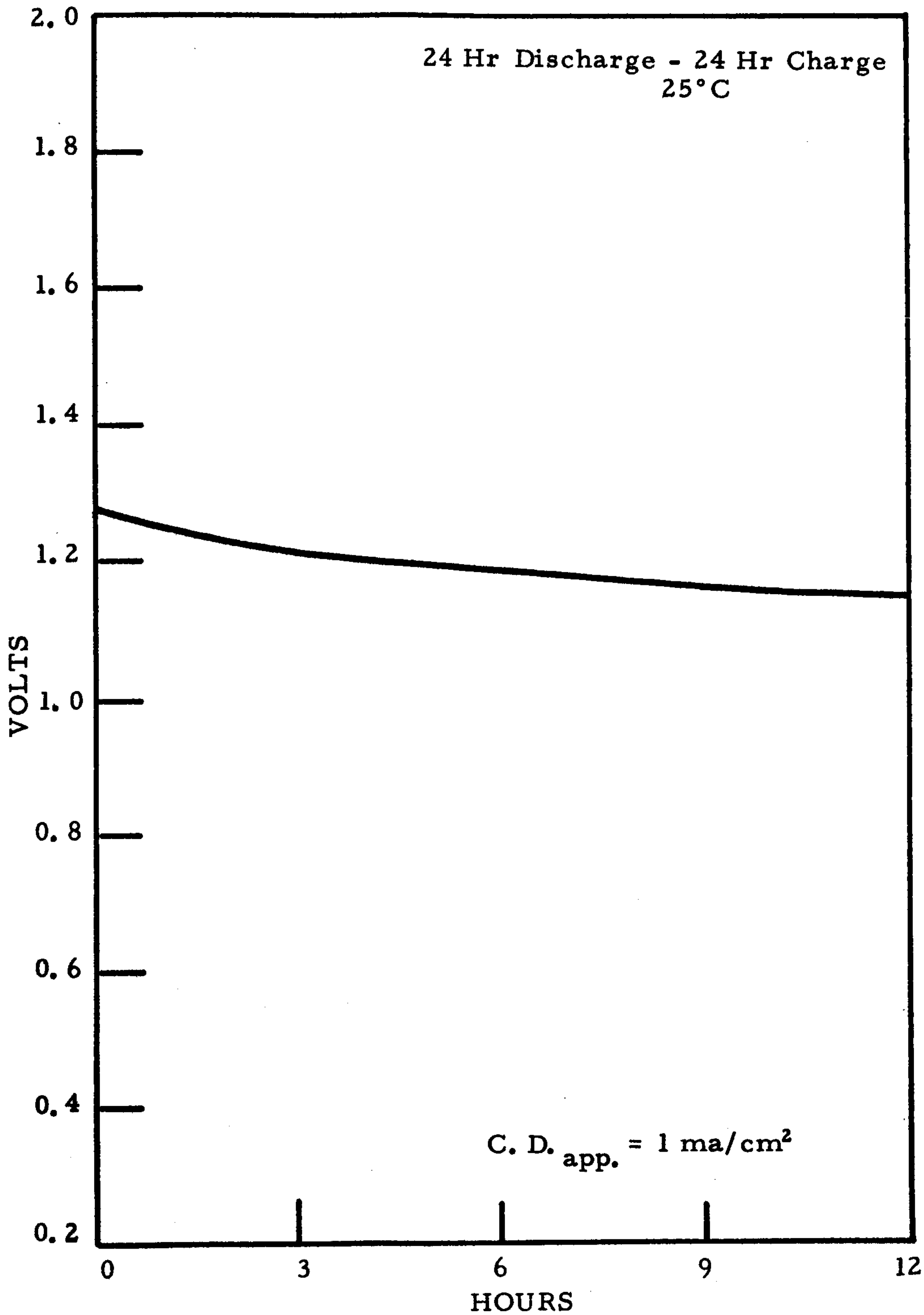

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FIGURE 13

RECHARGEABLE ZINC OXYGEN CELL
CYCLING 2 HOUR DISCHARGE/ 2 HOUR CHARGE

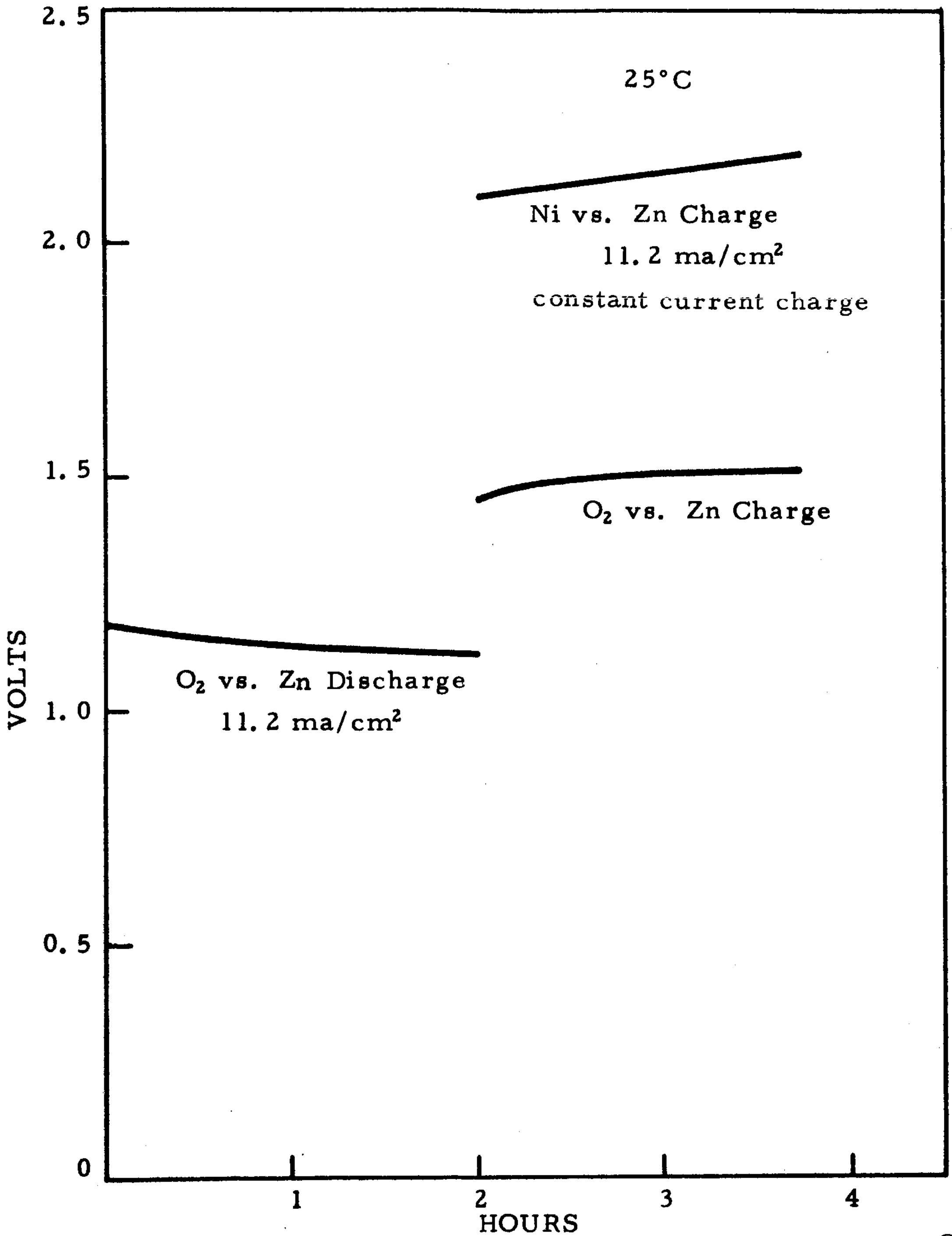

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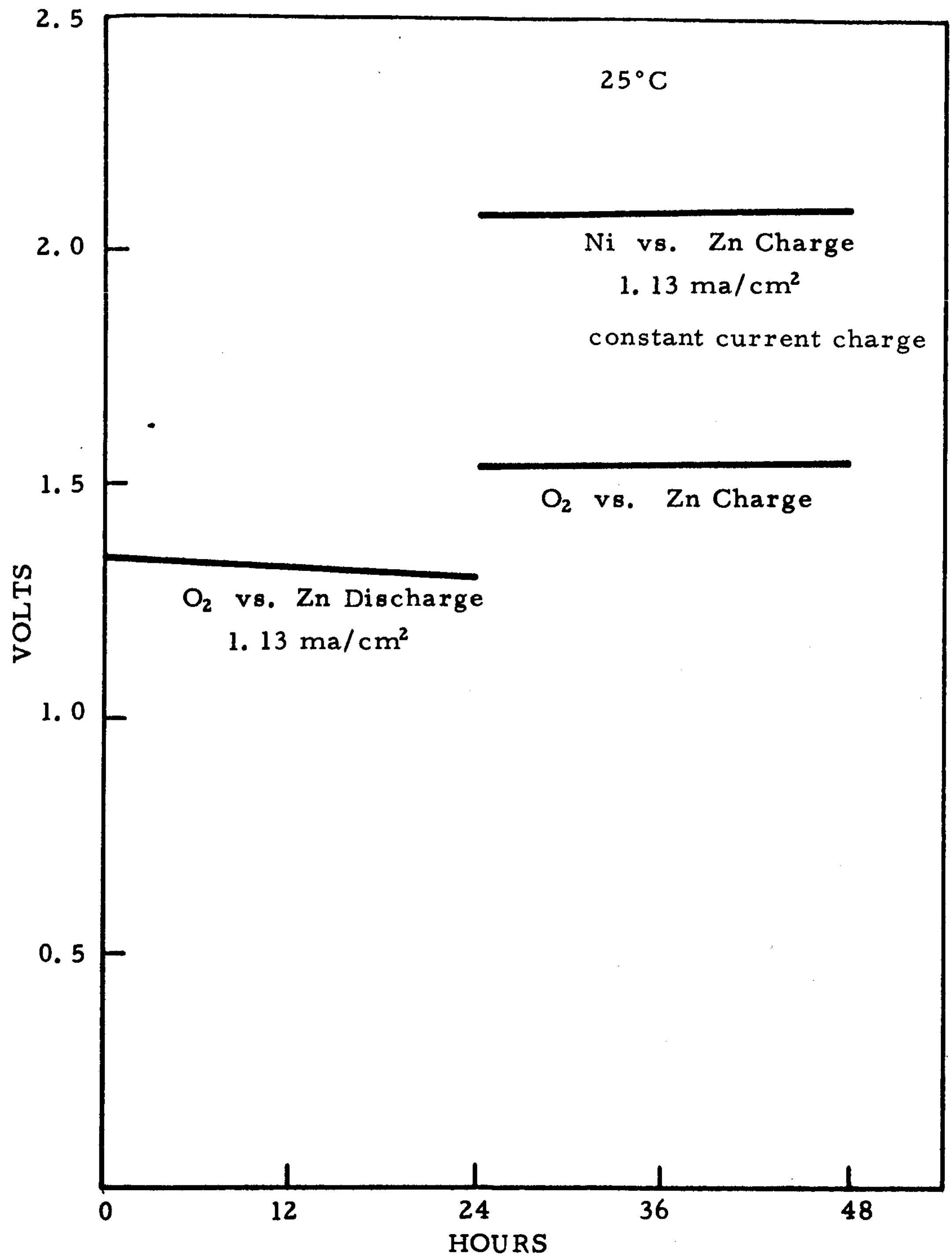
FIGURE 14

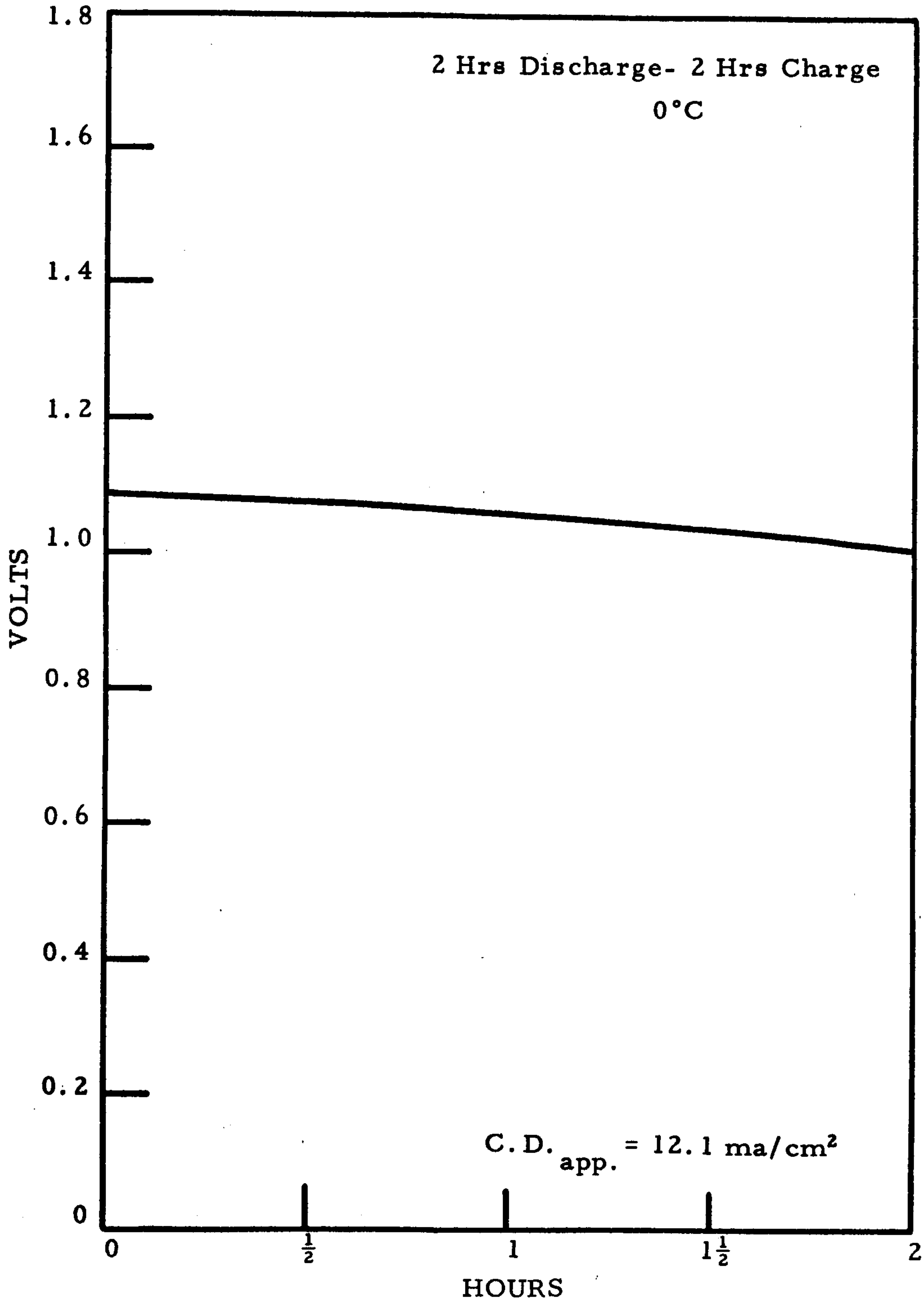
RECHARGEABLE ZINC-OXYGEN CELL
CYCLING 24 HOUR DISCHARGE/24 HOUR CHARGE



C-3455

FIGURE 15

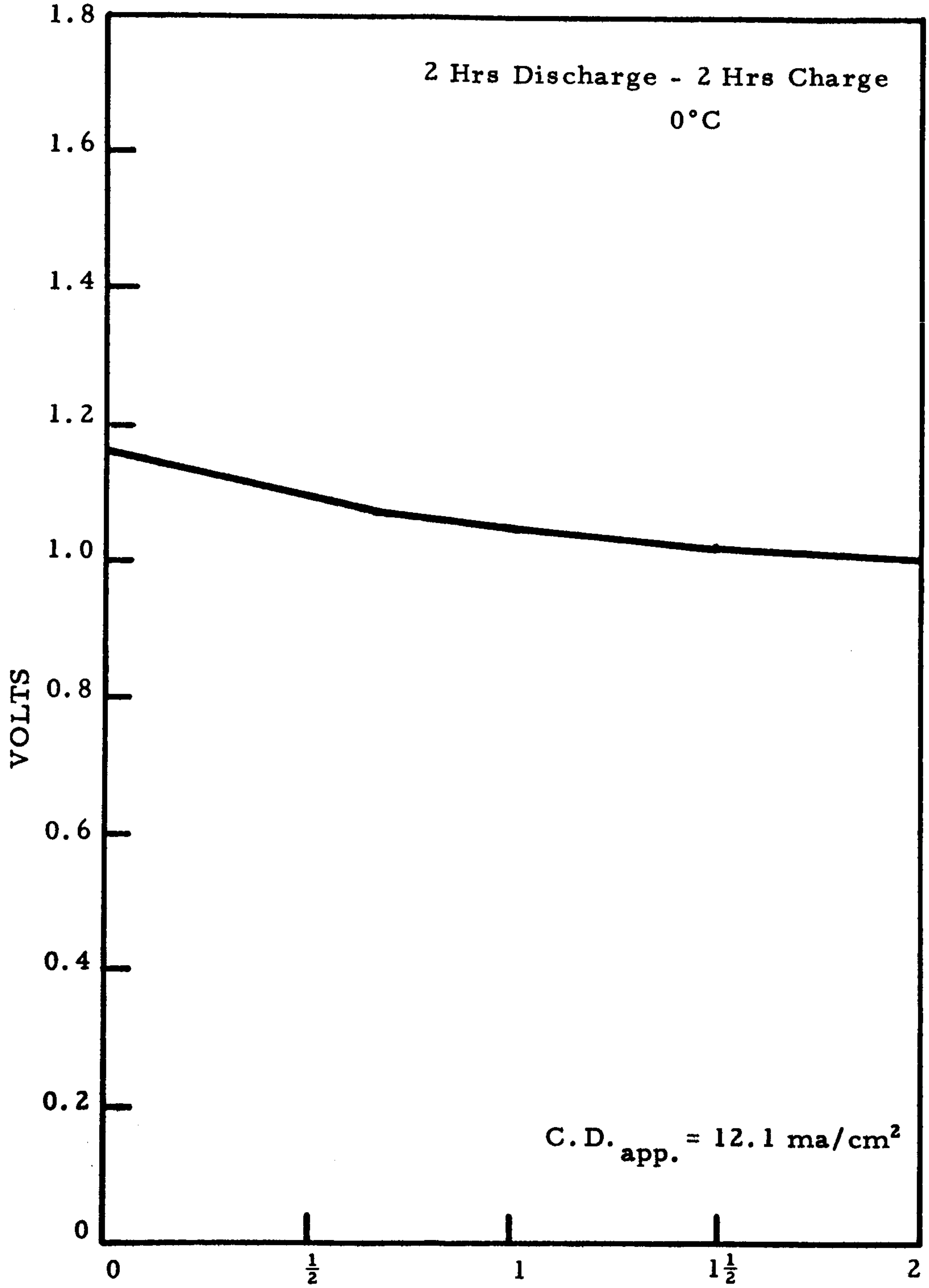
1ST DISCHARGE RECHARGEABLE $Zn-O_2$ UNIT CELL



C-3330

FIGURE 16

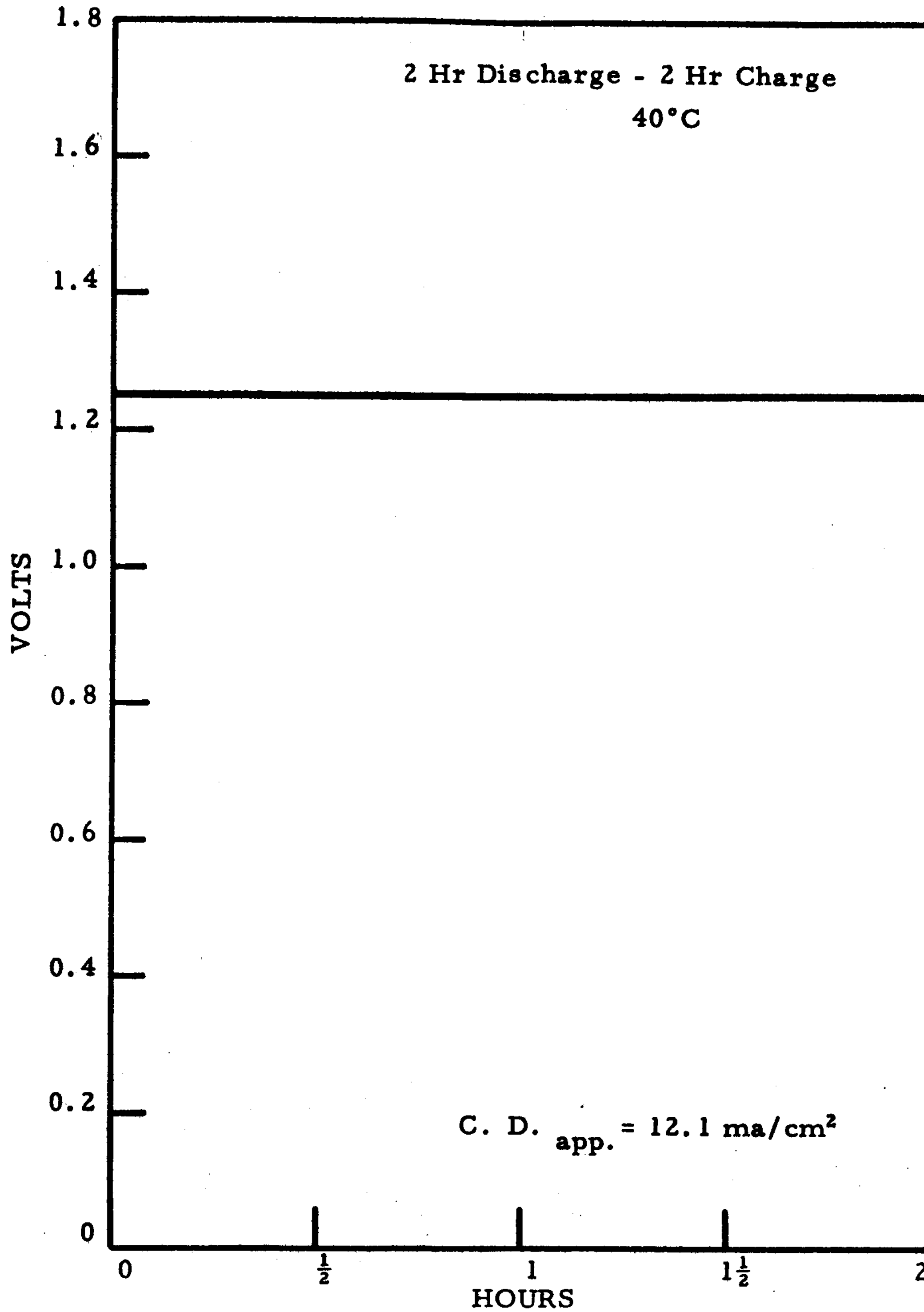
4TH DISCHARGE OF RECHARGEABLE $Zn-O_2$ UNIT CELL



C-3326

FIGURE 17

1ST DISCHARGE OF RECHARGEABLE $Zn-O_2$ UNIT CELL



C-3317

FIGURE 18

5TH DISCHARGE OF RECHARGEABLE $Zn-O_2$ UNIT CELL

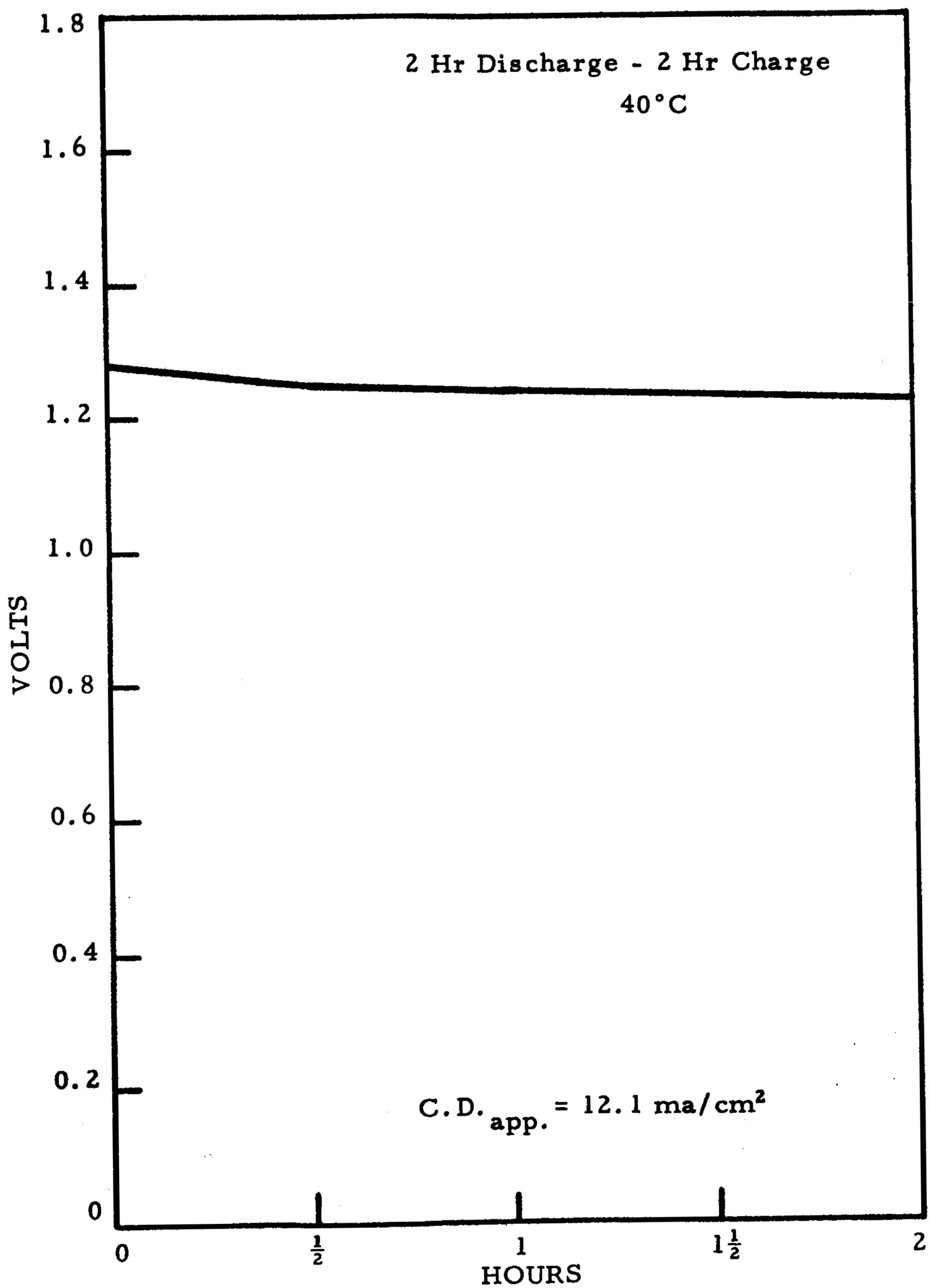


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FIGURE 19

9TH DISCHARGE OF RECHARGEABLE $Zn\text{-}O_2$ UNIT CELL

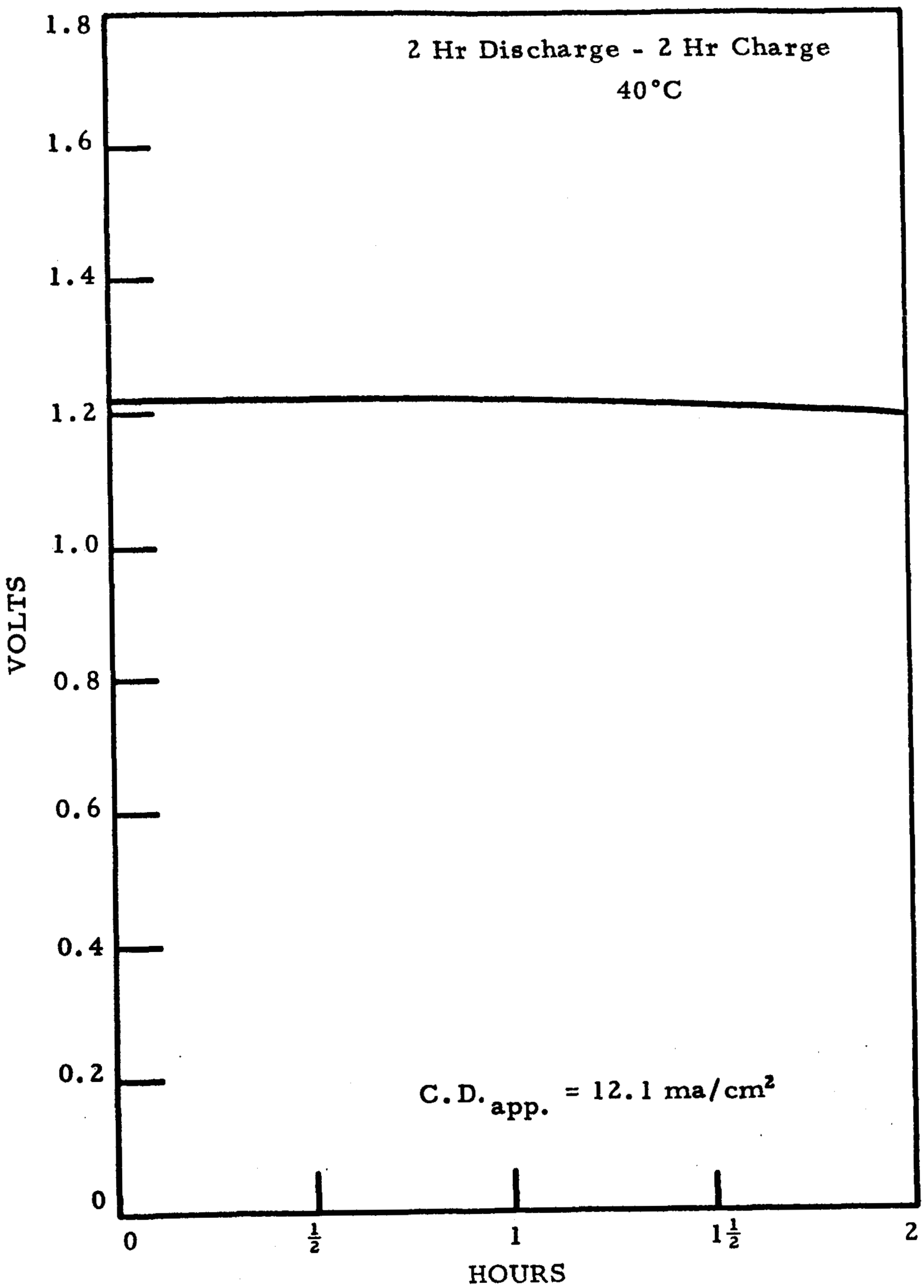

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FIGURE 20

COMPARISON OF UNIT CELL PERFORMANCE AT 0°, 25°, and 40°C
FIRST DISCHARGE

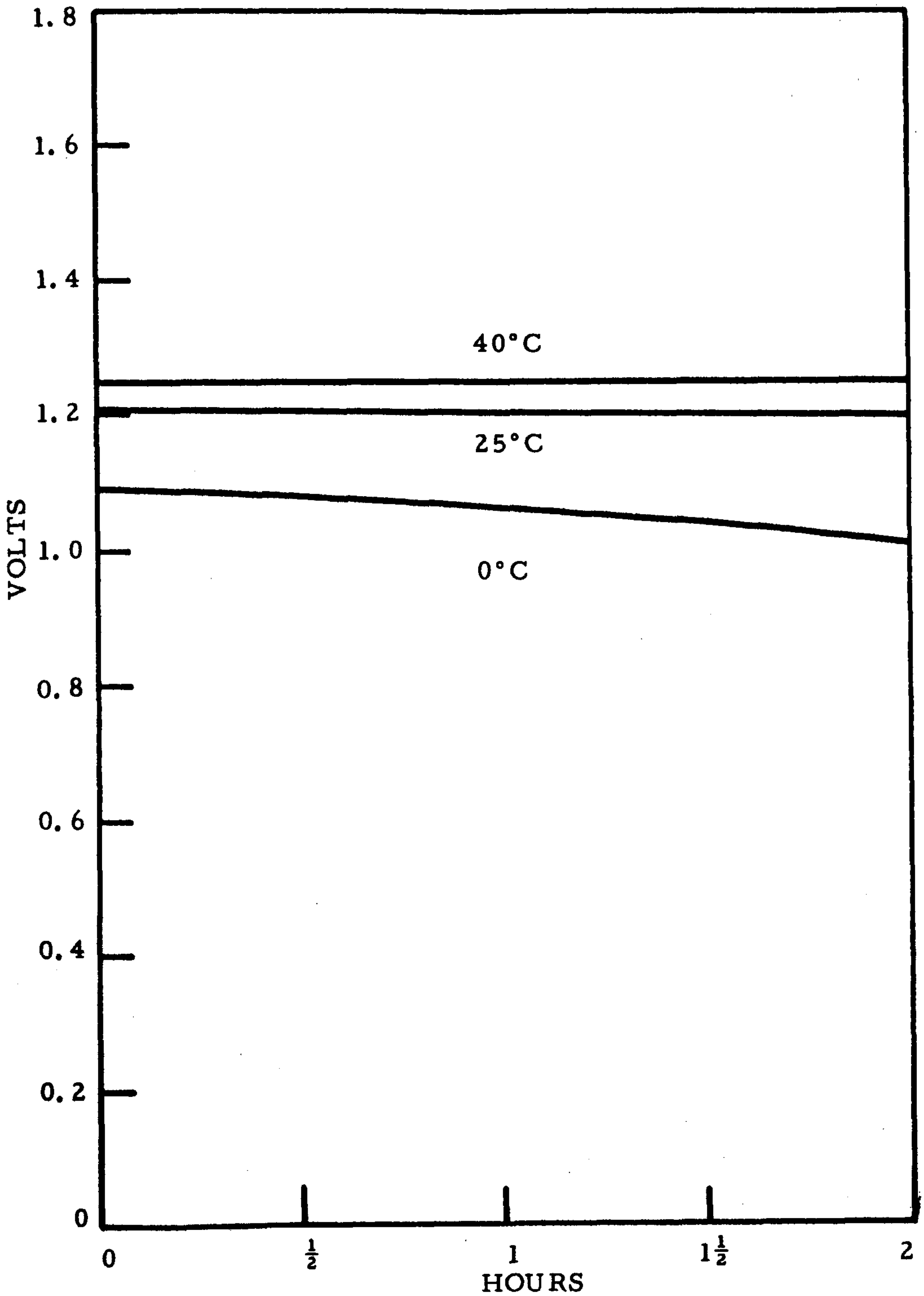

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FIGURE 21

COMPARISON OF UNIT CELL PERFORMANCE AT 0°, 25°, and 40°C
SECOND DISCHARGE

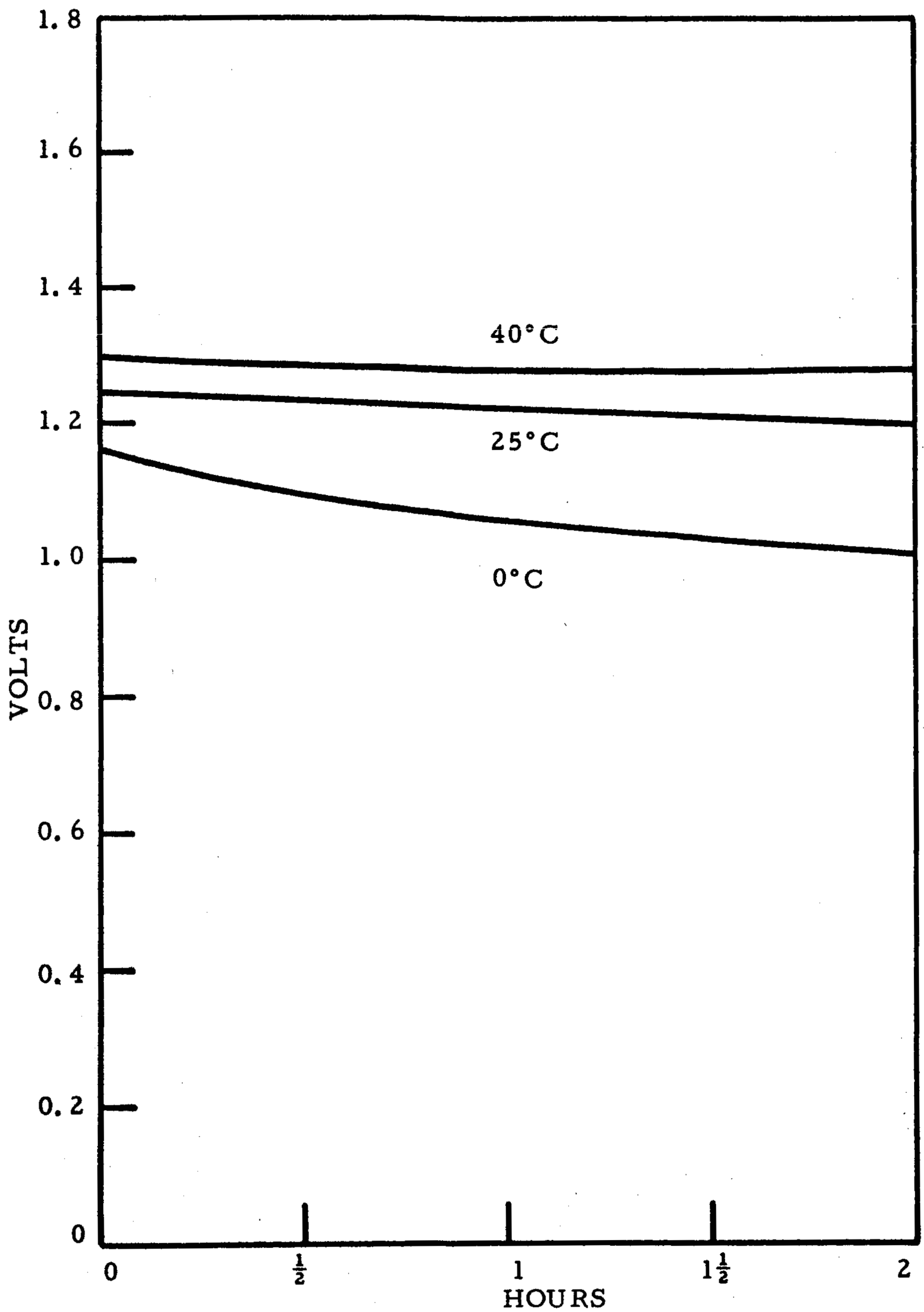

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FIGURE 22

COMPARISON OF UNIT CELL PERFORMANCE AT 0°, 25°, and 40°C
THIRD DISCHARGE

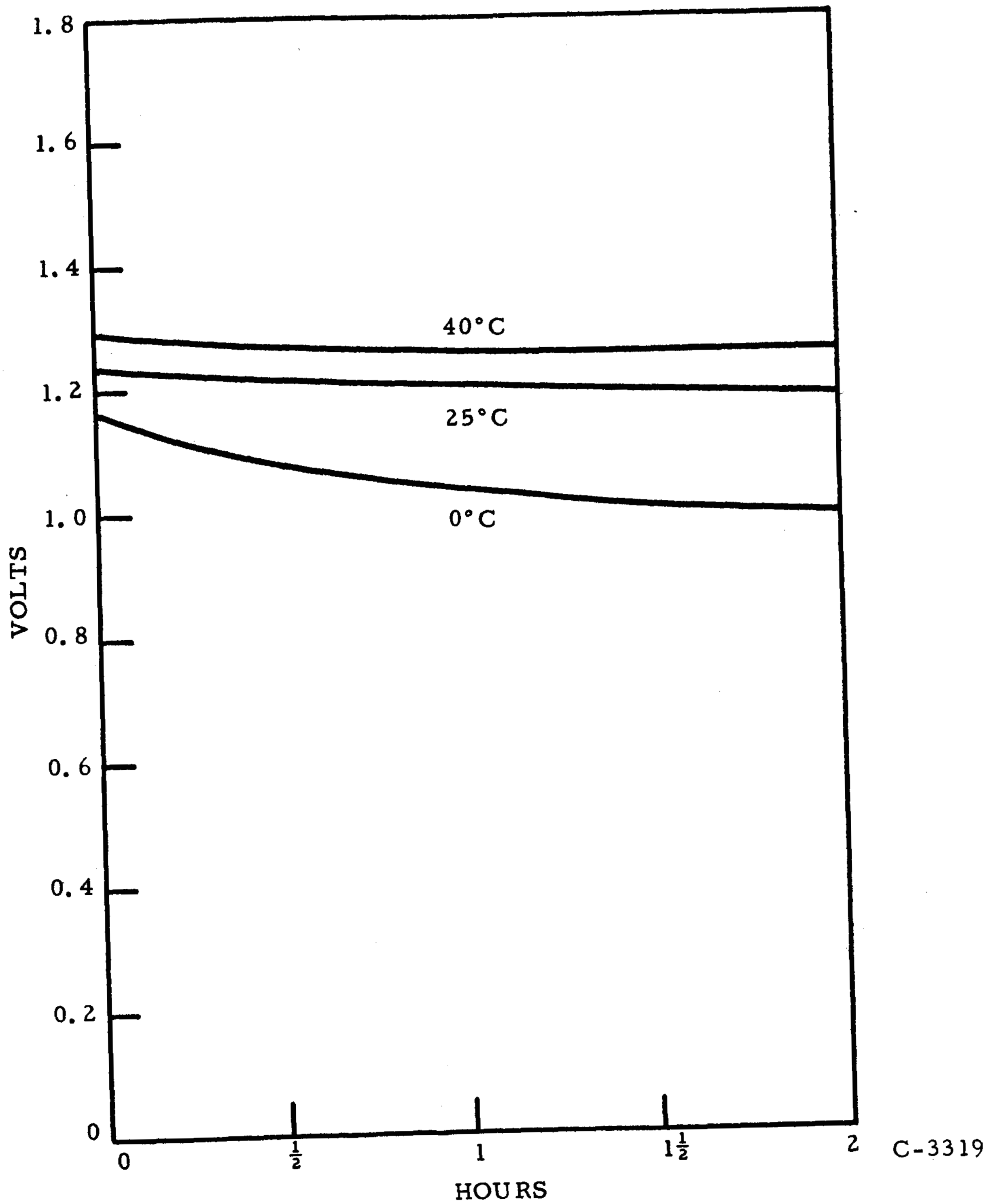
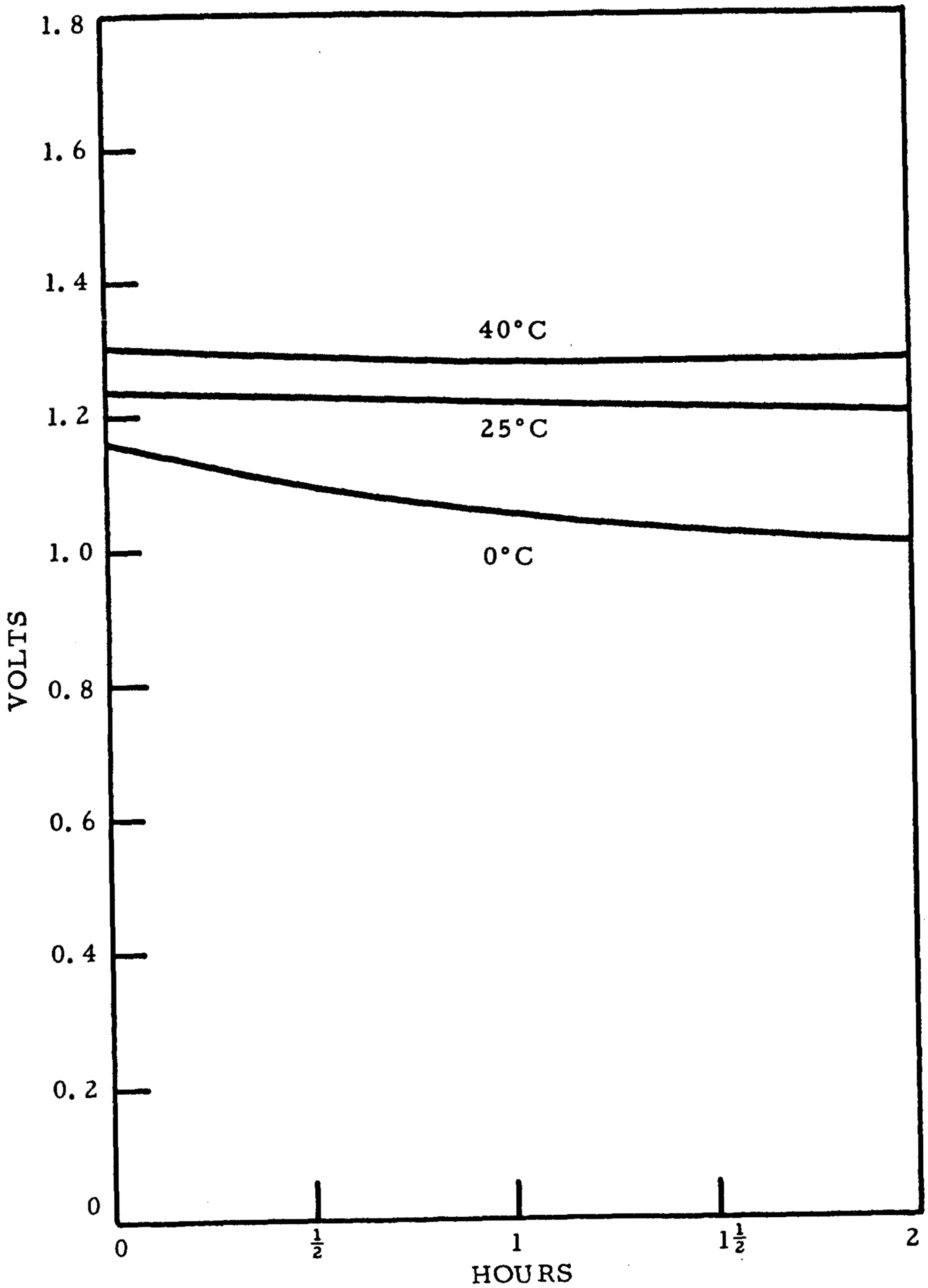


FIGURE 23

COMPARISON OF UNIT CELL PERFORMANCE AT 0°, 25°, and 40°C
FOURTH DISCHARGE



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